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Indian Standard

SODIUM PERCHLORATE SOLUTION FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

UDC 662·222·22

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 4

Explosives and Pyrotechnics Sectional Committee, CHD 026

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Sodium perchlorate is widely used in the form of solution in the manufacture of industrial explosives. It is a strong oxidizing agent. It has a molecular formula $NaClO_4$ and molecular mass of 122·5.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off values should be the same as that of the specified values in this standard.

Indian Standard

SODIUM PERCHLORATE SOLUTION FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for sodium perchlorate solution for use in explosives and pyrotechnic compositions.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
4905 : 1968	Methods for random sampling
1260 (Part 1) : 1986	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless liquid, free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in securely closed PVC containers or as agreed to between the purchaser and the supplier and subject to relevant provision of Red Tariff No. 17 of 1954 issued by the Indian Railway Conference Association, with any alterations or additions made thereafter.

4.1.1 Necessary safeguard shall be provided against risk of dangerous explosions which might occur when the material is heated or subjected to concussion or triturated with organic substances, charcoal, sulphur, sulphides, powdered metallic iron or other oxidizable substances, and precautions shall be taken at all times to prevent accidents by explosions.

4.2 Marking

The containers shall be legibly and indelibly marked with the following information:

a) Name of the material;
b) Mass of the material in the container;
c) Indication of the source of manufacture;
d) Month and year of manufacture; and
e) Lot or batch number in code or otherwise.

4.2.1 The containers may also be marked with the 'STANDARD MARK' of the Bureau.

4.2.2 Each container shall be marked with appropriate explosive symbol specified in IS 1260 (Part 1) : 1986.

Table 1 Requirements for Sodium Perchlorate Solution for Explosives Industry

(*Clause* 3.2)

Sl No.	Characteristics	Requirements	Method of Test, Ref to Cl No. in Annex A
(1)	(2)	(3)	(4)
i)	Assay (as $NaClO_4$), percent by mass, *Min*	55·0	A-2
ii)	Chlorides (as NaCl), percent by mass, *Max*	0·5	A-3
iii)	Chlorates (as $NaClO_3$), percent by mass, *Max*	0·65	A-4
iv)	Barium, percent by mass, *Max*	0·2	A-5
v)	Sulphates (as SO_4), percent by mass, *Max*	0·1	A-6
vi)	Specific gravity at 27°C, *Min*	1·45	A-7

5 SAMPLING

The procedure for drawing representative samples from a lot, the number of tests to be performed and criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(*Clause* 3.2 and *Table* 1)

METHODS OF TEST FOR SODIUM PERCHLORATE SOLUTION FOR EXPLOSIVES INDUSTRY

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF ASSAY (as $NaClO_4$)

A-2.1 Apparatus

The apparatus consists of a wide-necked flask (about 300 ml) fitted with a cork carrying a reflux condenser, an inlet tube (6 mm bore) reaching the bottom of the flask, and a short tube (at least 12 mm bore) fitted with a cork.

A-2.2 Reagents

A-2.2.1 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1976.

A-2.2.2 *Sulphur Dioxide* — gas.

A-2.2.3 *Dilute Sulphuric Acid* — approximately 12 N.

A-2.2.4 *Titanous Chloride Solution*

Approximately 3 percent, prepared by diluting 10 ml of 15 percent (*m/v*) titanous chloride solution to 50 ml with concentrated hydrochloric acid.

A-2.2.5 *Ammonium Thiocyanate Solution* — approx 0·1 N.

A-2.2.6 *Potassium Chlorate* — free from perchlorates.

A-2.2.7 *Standard Ferric Ammonium Sulphate Solution*

Approximately 0·1 N, prepared by dissolving 48·22 g of ferric ammonium sulphate in 500 ml of freshly boiled water that has been acidified with 10 ml of dilute sulphuric acid (40 percent by mass). Make up the volume to 1 000 ml with freshly boiled water (Solution *A*):

a) Measure into a 200-ml conical flask, 25 ml of freshly boiled water, 25 ml of 0·1 N potassium bichromate solution and 15 ml of dilute sulphuric acid (40 percent by mass). Fit the conical flask with a rubber bung bored with two holes, through one of which passes a leading tube, reaching about 50 mm into the flask. Pass a rapid stream of carbon dioxide into the flask for several minutes in order to displace the air. Place the jet of the burette containing the titanous chloride solution in the second hole of the rubber bung and, whilst continuing to pass carbon dioxide, run 20 ml of the titanous chloride solution. Mix the liquid by swirling the flask gently and titrate the excess of titanous salt with Solution A added from a 25 ml burette, the jet of which is placed inside the second hole in the rubber bung. Towards the end of the titration, that is, when the dark colour due to the titanous salt has nearly disappeared, add 10 ml of ammonium thiocyanate solution and continue the addition of Solution A until a pink colour is obtained, that is, permanent for one minute. Maintain a slow stream of carbon dioxide throughout the titration.

b) In the same way, titrate 20 ml of the titanous chloride solution with Solution A from a 50 ml burette but omitting 0·1 N potassium bichromate solution. From the volume of Solution A used in this titration, deduct the volume of ferric ammonium sulphate solution used in the first titration (this difference corresponds to 25 ml of 0·1 N potassium bichromate solution), and calculate the exact normality of ferric ammonium sulphate solution.

A-2.3 Procedure

Weigh accurately about 5 g of the material and transfer it completely into the wide-necked flask. Dissolve it in water and pass sulphur dioxide through the solution. Note the progress of the reduction by evolution of heat and the completion of reduction by the solution becoming cold (the time taken is generally 1 to 2 hours). Remove the excess of sulphur dioxide completely by boiling and by the aid of a current of carbon dioxide passed through the liquid. Complete removal is indicated by the absence of odour of sulphur dioxide. As a precaution, continue the boiling gently for 30 minutes after the odour has disappeared. After cooling, 20 ml of dilute sulphuric acid and dilute the solution with water to 150 ml. Fit the flask to a reflux condenser and pass carbon dioxide through the liquid continuously until the end of the titration. Boil the solution for a few minutes to remove dissolved air. Add an exact volume (say, 10 ml) of titanous chloride solution by means of a pipette through the wide glass tube which is immediately closed with a small cork. Continue refluxing for one hour, after which remove the flask from the condenser and cool in water. Add 5 ml of ammonium thiocyanate solution, and then titrate with Solution A through the glass-lined hole in the cork (Fig. 1).

A-2.3.1 Carry out a blank determination using all reagents used in the test under **A-2.3** but without sample.

A-2.3.2 Apparatus for titration in inert (carbon dioxide) atmosphere and for preservation and addition of titanous chloride solution are shown in Fig. 1 and 2 respectively.

A-2.4 Calculation

$$\text{Potassium perchlorate (as } KClO_4 \text{), percent by mass, } Min = 1{\cdot}73 \times \frac{(V_1 - V_2)\,N}{M}$$

where

V_1 = volume in ml of standard ferric ammonium sulphate solution required for the blank determination (*see* **A-2.3.1**),

V_2 = volume in ml of standard ferric ammonium sulphate solution (*see* **A-2.3**) required for test with the material,

N = normality of standard ferric ammonium sulphate, and

M = mass in g of the material taken for the test.

A-3 DETERMINATION OF CHLORIDES

A-3.1 Reagents

A-3.1.1 *Standard Silver Nitrate Solution* — 0·1 N approximately.

A-3.1.2 *Concentrated Nitric Acid*

A-3.1.3 *Ferric Ammonium Sulphate Indicator* — 10 percent (*m/v*).

A-3.1.4 *Standard Ammonium Thiocyanate Solution* — 0·1 N approx.

A-3.1.5 *Nitrobenzene*

A-3.2 Procedure

Weigh accurately 2·0 g of the material and mix it with about 50 ml of water. Add 10 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 5 ml of nitrobenzene. Shake vigorously for one minute. Add 2 ml of ferric ammonium sulphate indicator solution and titrate excess silver nitrate with standard ammonium thiocyanate solution shaking well between successive additions to the first persistent colour change.

A-4 DETERMINATION OF CHLORATES

A-4.1 Reagents

A-4.1.1 *Ferric Sulphate or Ferrous Ammonium Sulphate Solution* — Approximately 0·1 N.

A-4.1.2 *Standard Potassium Permanganate Solution* — 0·1 N.

A-4.1.3 *Manganous Sulphate Solution* — Dissolve 10 g of the material in water and dilute to 100 ml.

A-4.1.4 *Dilute Sulphuric Acid* — Approximately 2 N.

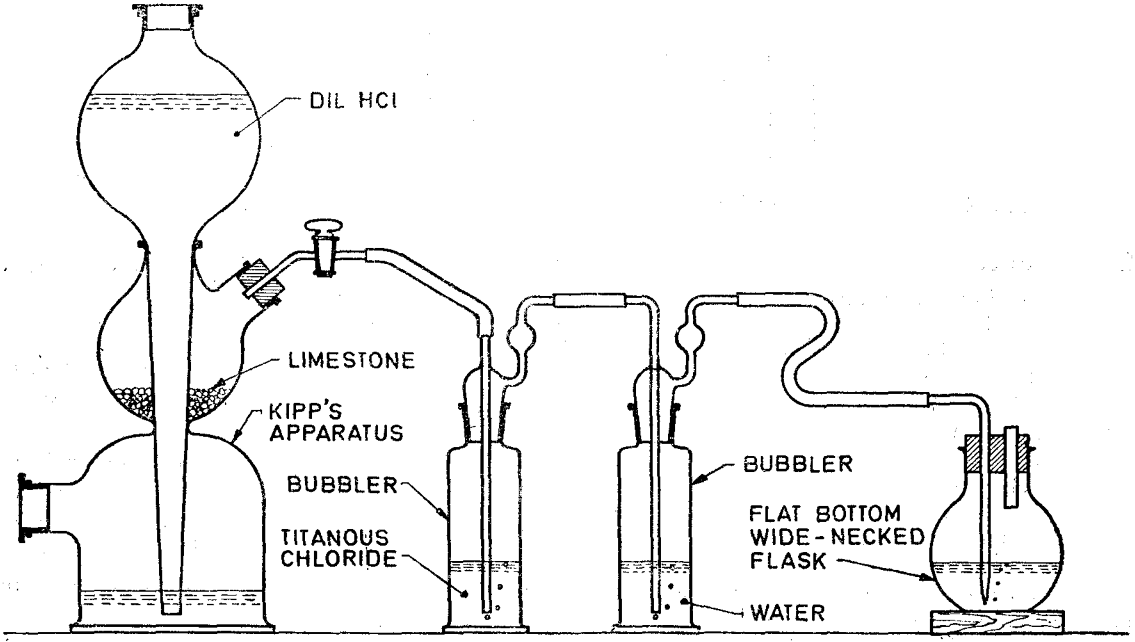

FIG. 1 APPARATUS FOR TITRATION IN AN INERT (CARBON DIOXIDE) ATMOSPHERE

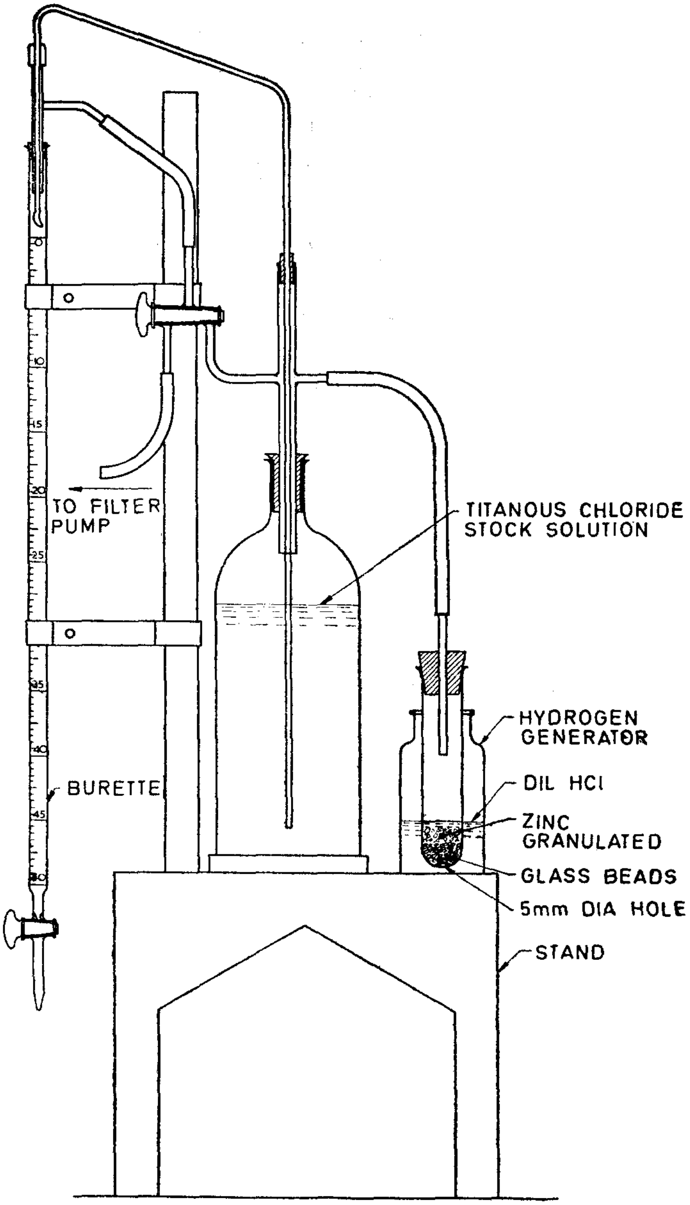

FIG. 2 APPARATUS FOR PRESERVATION AND ADDITION OF TITANOUS CHLORIDE SOLUTION

A-4.2 Procedure

Dry the material over sulphuric acid for 24 hours. Weigh accurately about 0·10 g and mix it with 20 ml of water in a 250 ml conical flask. Add exactly 35 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid, close the flask with a valve stopper to prevent access of air and boil for 15 minutes. Cool the contents of the flask, add 10 ml of manganous sulphate solution and titrate the excess of ferrous sulphate against standard potassium permanganate. Run a blank in the same manner with 35 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid. The difference between the volume of potassium permanganate consumed in the blank and the sample represents the chlorate content.

A-4.3 Calculation

$$\text{Chlorate (as } NaClO_3 \text{)} = 2{\cdot}043 \, \frac{(V_2 - V_1)\,N}{M}$$

where

V_2 = volume in ml of permanganate solution consumed in the blank experiment,

V_1 = volume in ml of permanganate solution consumed by excess of reducing agent,

N = normality of permanganate solution, and

M = mass in g of the material taken for the test.

A-5 DETERMINATION OF BARIUM

A-5.0 Outline of the Method

Barium is precipitated as barium chromate which is then dissolved in dilute hydrochloric acid and treated with solid potassium iodide. The liberated iodine is titrated against standard sodium thiosulphate solution.

A-5.1 Reagents

A-5.1.1 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-5.1.2 *Concentrated Hydrochloric Acid* — *See* IS 265 : 1976.

A-5.1.3 *Concentrated Ammonium Hydroxide* — 20 percent (*m/m*).

A-5.1.4 *Ammonium Chloride* — solid.

A-5.1.5 *Hydrogen Sulphide* — gas.

A-5.1.6 *Lead Acetate Paper*

A-5.1.7 *Methyl Orange Indicator*

Dissolve 0·1 g of methyl orange in 100 ml of water.

A-5.1.8 *Glacial Acetic Acid* — *See* IS 695 : 1975.

A-5.1.9 *Ammonium Acetate Solution* — 30 percent (*m/v*).

A-5.1.10 *Ammonium Chromate Solution* — 10 percent (*m/v*), neutral to methyl orange.

A-5.1.11 *Ammonium Chromate Solution* — 1 percent (*m/v*), neutral to methyl orange.

A-5.1.12 *Ammonium Carbonate Solution* — approx 20 percent (*m/v*).

A-5.1.13 *Silver Nitrate Solution* — approx 2 percent (*m/v*).

A-5.1.14 *Dilute Nitric Acid* — approximately 2 N.

A-5.1.15 *Potassium Iodide* — solid.

A-5.1.16 *Standard Sodium Thiosulphate Solution* — 0·1 N.

A-5.1.17 *Starch Indicator Solution*

A-5.2 Procedure

Weigh accurately about 0·25 g of the material and mix it with about 30 ml of dilute hydrochloric acid, add 100 ml of water, boil and filter. Wash the residue with water. Collect the filtrate and the washings in a 500-ml beaker and make it alkaline with concentrated ammonium hydroxide. Add about one gram of ammonium chloride, pass hydrogen sulphide gas and filter. Pass hydrogen sulphide gas through the filtrate again till no turbidity appears. Boil the filtrate to drive away hydrogen sulphide gas and test for complete removal of the gas by lead acetate paper. Neutralize the solution with dilute hydrochloric acid using methyl orange indicator. Add 5 to 6 drops of glacial acetic acid together with ammonium acetate solution to neutralize any free mineral acid. Heat the solution and add an excess of ammonium chromate solution.

A-5.2.1 Allow the precipitate of a barium chromate to settle for an hour. Filter through a filter paper and wash with one percent ammonium chromate solution until the filtrate is free of soluble salts which are tested if a portion of the filtrate produces no cloudiness with ammonium hydroxide and ammonium carbonate. Finally wash the residue free of ammonium chromate with water till a portion of the filtrate gives only slight reddish brown colour with silver nitrate solution.

A-5.2.2 Pierce the filter paper containing the barium chromate precipitate and wash the precipitate into a beaker with hot dilute nitric acid. Dilute the solution to about 200 ml and boil. Add ammonium acetate solution to neutralize any free nitric acid. Heat and add sufficient ammonium chromate solution. Filter and repeat washings as in **A-5.2.1** above. Dissolve the precipitate of barium chromate finally in 50 to 100 ml of dilute hydrochloric acid, add about 2 g of solid potassium iodide and allow to react for ten minutes. Titrate the liberated iodine with standard sodium thiosulphate, using starch indicator.

A-5.3 Calculation

$$\text{Barium, percent by mass} = \frac{0{\cdot}457\,9 \times V}{M}$$

where

V = volume in ml of standard sodium thiosulphate required for the titration, and

M = mass in g of the material taken for the test.

A-6 DETERMINATION OF SULPHATES

A-6.1 Reagents

A-6.1.1 *Concentrated Hydrochloric Acid*

A-6.1.2 *Barium Chloride Solution* — approx 10 percent (*m/v*).

A-6.2 Procedure

Weigh accurately about 2·0 g of the sample and mix it with 50 ml of water. Add 15 ml of concentrated hydrochloric acid and dilute to 200 ml with water. Filter, if necessary. Heat to boiling, add drop by drop, with constant stirring, 10 ml of barium chloride solution and allow to stand for about 2 hours. Filter through a tared sintered glass crucible (G. No. 4), wash the precipitate thoroughly with hot water till free from chlorides and dry between 105 to 100°C to constant mass.

A-6.3 Calculation

$$\text{Sulphates (as } Na_2SO_4 \text{), percent by mass} = \frac{60 \cdot 92\, M_1}{M}$$

where

M_1 = mass in g of the barium sulphate precipitate, and

M = mass in g of the material in the aliquot taken for the test.

A-7 DETERMINATION OF SPECIFIC GRAVITY

A-7.1 Apparatus

A-7.1.1 *Specific Gravity Bottle*

Closed type, made of glass and of a size and type suitable for use with the material under test.

A-7.1.2 *Water Bath*

Thermostatically controlled with an accuracy of ± 0·5°C.

A-7.2 Procedure

A-7.2.1 Clean and dry the specific gravity bottle and weigh it with its stopper to the nearest milligram. Fill the bottle with water at 27°C ± 0·5°C and determine the apparent mass of the contents.

A-7.2.2 Clean and dry the specific gravity bottle, fill it with the sodium perchlorate solution and determine, in a similar manner the apparent mass of the contents.

NOTE — Ensure that the temperature of the sodium perchlorate solution, water and the specific gravity bottle does not change during the test by keeping them in the constant temperature bath.

A-7.3 Calculation

Calculate the specific gravity of the sample at 27°C from the following formula and report the results correct to 2 places of decimal:

$$\text{(} NaClO_4 \text{ solution) } 27°C = \frac{(M_1 + A)}{(M_2 + A)} \times wt$$

where

M_1 = apparent mass in g of the sodium perchlorate solution required to fill the bottle at 27°C,

M_2 = apparent mass in g of the water required to fill the bottle at 27°C,

wt = density of water at 27°C,

A = buoyancy correction ($at \times M_2$), and

at = density of air at 27°C and 760 mm Hg pressure = 0·001 177 g/ml.

ANNEX B

(*Clause* 5)

SAMPLING OF SODIUM PERCHLORATE SOLUTION

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Necessary safeguards as prescribed in **4.1.1** shall be provided in taking out material from the containers for sampling.

B-1.2 The samples and the sampling implement shall be well protected from any possible contamination.

B-1.3 The sampled material shall be placed in suitable, dry, clean container which shall be marked with all relevant details after filling.

B-1.4 Samples shall be taken from different parts of the selected containers with the help of suitable sampling implements.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers of sodium perchlorate manufactured at a time and under similar conditions of production shall be grouped together to constitute a lot. Each lot shall be tested separately for testing the various requirements of the specification.

B-2.2 The number of containers to be selected for the sample shall be as given in Table 2.

Table 2 Scale of Sampling

Number of Containers in a Lot	Number of Containers to be Selected
(1)	(2)
Up to 25	3
26 ,, 50	4
51 ,, 100	5
101 and above	7

These containers shall be selected at random from the lot as per IS 4905 : 1968.

B-2.3 Preparation of Test Sample

From each of the selected containers a representative portion of material shall be taken, which shall be sufficient for carrying out tests for all characteristics given in the specification.

B-2.3.1 *Composite Sample*

Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to make a composite sample.

B-2.3.2 *Individual Sample*

The remaining portion of the material from each selected container shall constitute an individual test sample.

B-2.4 Number of Tests

Tests for description and assay (as $NaClO_4$) shall be conducted on the individual sample whereas tests for all other characteristics shall be conducted on the composite sample.

B-2.5 Criteria for Conformity

The lot shall be deemed to conform to the specifications if all the test results according to **B-2.4** satisfy the corresponding requirements.

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